

Company, Inc. (Techalloy), Union, Illinois. The Consent Order was signed by U.S. EPA and Techalloy on 27 January 1993. The five SWMUs are listed below:

- Wire slag disposal area.
- BF-5 oil drums.
- Spent acid holding pond.
- Plating wastewater disposal area.
- Concrete evaporation pad.

The locations of these five units are depicted in Figure 2-1.

The Phase I activities are designed to determine the vertical and horizontal extent and concentrations of constituents in the surrounding soil and groundwater at each of the SWMUs in question.

The Phase I source characterization activities will consist of the advancement of soil borings in and around each SWMU area. The borings will be advanced through the vadose zone soils to determine the presence, nature, magnitude, and lateral and vertical extent of constituents potentially related to each SWMU in question. The determination of the number of borings to be advanced in and around each SWMU area was based on the size of the SWMU, the activities associated with it and the proximity to the sampling areas around adjacent SWMUs. Borings have been located within the active area of each SWMU to determine the potential impact to the soil. Borings have been located outside the perimeter of each SWMU area in each potential direction of migration to determine the potential lateral and vertical extent of any impact. The borings will be advanced above the water table which lies at approximately 9 feet below ground surface (bgs). Two soil samples will be collected from each boring to allow visual classification of the soils. Two soil samples will also be collected for laboratory analysis at each boring location to determine the vertical distribution of constituents in the vadose zone soils. The samples will be collected from 1 to 2 feet below the native soil surface and 5 to 6 feet bgs, above the capillary fringe of the water table.

To advance the soil borings, Roy F. Weston, Inc. (WESTON®) will employ the use of a Geoprobe hydraulic press. The Geoprobe unit provides rapid advancement of borings and

- Define the problem.
- Assign responsibility for investigating the problem.
- Determine a corrective action to eliminate the problem.
- Assign and accept responsibility for implementing the corrective action.
- Establish effectiveness of the corrective action and implement the correction.
- Verify that the corrective action has eliminated the problem.

The initial responsibility to monitor the quality of a function or analytical system lies with the individual performing the task or procedure. Quality indicators are evaluated against laboratory-established or client-specified QA/QC requirements. If the assessment reveals that any of the QC acceptance criteria are not met, the analyst must immediately assess the analytical system to correct the problem. Figure 14-1 presents WESTON-Gulf Coast Laboratories, Inc. Corrective Action Documentation Form. When an acceptable resolution cannot be met and/or data quality is negatively impacted, the analyst will notify the appropriate supervisor and initiate a Sample Discrepancy Report Form (Figure 14-2).

When the appropriate corrective action measures have been defined and the analytical system is determined to be "in control" or the measures required to put the system "in control" have been identified and scheduled, the problem and resolution or planned action is documented in the appropriate notebook. If a Sample Discrepancy Report (SDR) was required, the report will be routed for proper authorizations and signatures. Depending on the nature of the SDR, the project manager or section manager will sign the SDR for notification that the discrepancy has been documented and corrected.

Data generated concurrently with an out-of-control system will be evaluated for usability in light of the nature of the deficiency. If the deficiency does not impair the usability of the results, data will be reported and the deficiency noted in the case narrative. Where sample results are impaired, the Laboratory Project Manager is notified by a written Sample Discrepancy Report and appropriate corrective action (e.g., re-analysis) is taken and documented.

## **SECTION 15**

### **QUALITY ASSURANCE REPORTS TO MANAGEMENT**

WESTON's Project Manager and Quality Assurance Manager will audit the use of this QAPP. The preparation of a QA Report is not anticipated except as necessitated by problems arising during the project. If these problems should require the preparation of a QA Report, this task will be the responsibility of WESTON's Project Manager. The report may also include an assessment of field activities, data quality and the results of system and/or performance audits, as applicable. Any QA Report prepared by WESTON's Project Manager will be submitted to WESTON's Project Director, Techalloy's Project Director, and U.S. EPA's RCRA Project Coordinator. The QA report may be incorporated into a monthly progress report. The QA report will address any data validation or assessment that has taken place since the previous report. The final project report will include QA information, regardless of whether or not QA problems are observed.



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**Table 2-1**

**Objectives of Facility Investigation  
 Techalloy Site  
 Union, Illinois**

Objectives	Task	Activity	Purpose
<b>Phase I</b>			
Source Characterization	Sample soil.	Logging of soils Sample collection/analysis	Define media. Determine existing concentrations and extent of constituents.
Pathway Characterization	Sample groundwater.	Sample collection/analysis	Determine vertical extent of constituents at individual SWMUs.
	Confirm groundwater flow conditions on and off site.	Monitoring well water level measurements	Confirm groundwater migration flow path on and off site.
Extent of Volatile Constituent Migration	Collect groundwater samples downgradient of suspect source area, on and off site.	Groundwater samples from on- and off-site monitoring wells	Confirm existing level and extent of constituents in groundwater. Aid in potential placement of additional monitoring wells.
<b>Phase II</b>			
Source Characterization	Sample soil.	Logging of soils Sample collection/analysis	If extent not fully determined under Phase I, Phase II will define media and determine existing concentrations and extent of constituents.
Pathway Characterization	Completed under Phase I.		
Extent of Volatile Constituent Migration	Completed under Phase I.		

### 13.2.3 Completeness

The data completeness of laboratory analyses results will be assessed for compliance with the amount of data required for decisionmaking. Data completeness will be calculated using Equation 13-4.

$$\text{Completeness} = \frac{\text{Valid Data Obtained}}{\text{Total Data Planned}} \times 100 \quad \text{Equation 13-4}$$

### 13.2.4 Sensitivity

The achievement of method detection limits depend on instrumental sensitivity and matrix effects. Therefore, it is important to monitor the instrumental sensitivity to ensure the data quality through constant instrument performance. The instrumental sensitivity will be monitored through the analysis of method blank, calibration check samples, laboratory controlled sampling, the low concentration calibration standards, etc., in accordance with the requirements of the analytical SOPs (Appendix D).

All problems and corrective actions will be documented in the field logbook by the Field Team Leader. No field team member will start corrective action without prior communication of findings through the proper channels. If corrective actions are insufficient, the Field Team Leader may stop work following instructions from WESTON's Project Manager or the U.S. EPA's RCRA Project Coordinator.

#### **14.2 LABORATORY CORRECTIVE ACTIONS**

The laboratory-action protocols discussed in this section pertain to WESTON-Gulf Coast Laboratories, Inc. If any additional or alternate laboratories are assigned to perform analysis under this QAPP, WESTON will address changes to the corrective action procedure in an addendum to the QAPP. WESTON will submit the addendum to the U.S. EPA for review.

Laboratory corrective action may be immediate or long-term. Immediate corrective action to correct or repair nonconforming equipment and systems is generally done as the result of QC procedures. Any time an out-of-control situation occurs in the laboratory, a corrective action report (CAR) is completed by the analyst and submitted to the Unit Leader or Section Manager. The CARs document the out-of-control situation as well as the return-to control status. Original CARs are filed with the laboratory raw data or spreadsheets for future reference.

Long-term corrective action is generally undertaken due to QA issues that are most often identified during audits. Long-term corrective actions involve a deeper investigation into the root cause of the nonconformance, and may take much longer to identify and resolve. Staff training, method revision, replacement of equipment, LIMS reprogramming, etc., may be indicated long-term corrective actions.

All corrective actions, whether immediate or long-term, will employ the following steps to ensure a closed-loop corrective action system.



WESTON's Project Manager of the problem and anticipated change, and implementing the change.

If it is determined that the situation warrants a reportable nonconformance requiring corrective action, then WESTON's Project Manager will issue a nonconformance report. WESTON's Project Manager will be responsible for informing WESTON's Project Director, Techalloy's Project Director, and the U.S. EPA's RCRA Project Coordinator. WESTON's Project Manager will be responsible for making sure that corrective action for nonconformance are performed as follows:

- Evaluating all reported nonconformance.
- Controlling additional work on nonconforming items.
- Determining disposition or action to be taken.
- Maintaining a log of nonconformance.
- Reviewing nonconformance reports and corrective actions taken.
- Ensuring nonconformance reports are included in the final site documentation in project files.

If appropriate, the Project Manager will ensure that no additional work that is dependent on the nonconforming activity is performed until the corrective actions are completed.

All changes will be evaluated based on the potential to impact the quality of data. WESTON's Project Manager has ultimate responsibility for all site activities and must approve all changes verbally or in writing prior to field implementation by the Field Team Leader. WESTON's Project Director, Techalloy's Project Director, and the U.S. EPA's RCRA Project Coordinator will be notified when changes are implemented in the field.

and the measurement steps, to evaluate matrix effects. Table 9-1 lists the inorganic indicators. Table 9-2 lists the organic indicators.

### 9.3.1 Method Performance QC Indicators

- Preparation Batch - Most samples to be analyzed in the laboratory require some pretreatment before a measurement can be made. The necessary pretreatment may include extraction, digestion, distillation, etc. During the pretreatment step, samples are arranged into discrete, manageable groups, called preparation (prep) batches, to aid and control uniform treatment for all samples. Each prep batch will have a maximum of 20 investigative samples of the sample matrix (e.g., soil or water). In addition, the analyst adds QC indicators such as blanks, spikes, and duplicates to each prep batch to monitor the performance of the system. The laboratory will carry all QC associated with a preparation batch through the entire analytical procedure, from preparation to final analysis.
- Preparation Blanks - The laboratory uses the preparation blank (PB), also known as a method blank (MB) or reagent blank, to monitor potential contamination from the sample preparation process. The analyst will prepare preparation (prep) blank by processing a volume of deionized laboratory water for water samples, or a purified solid matrix for soil/sediment samples (when available), through the entire analytical scheme. The reagent blank volume must be approximately equal to the sample volumes being processed. Results will be calculated based on starting with a "blank" soil approximately equal to the weight of the samples.

For metals, the prep blank will be prepared by processing a volume of deionized laboratory water for both water (PBW) and soil or sediment (PBS) samples through the entire analysis scheme. Final results will be calculated as  $\mu\text{g/L}$  for the PBW. To facilitate comparison to the actual field samples, final results for the PBS will be calculated as  $\text{mg/kg}$  or  $\mu\text{g/kg}$ , assuming 100 percent solids and a weight equivalent to the aliquot used for the corresponding investigative samples.

- Laboratory Control Samples and Blank Spikes - Laboratory control samples and blank spikes are equivalent by definition. Laboratory control sample (LCS) is the terminology that the inorganics group uses, while blank spike (BS) is the terminology that the organics group uses. The LCS or BS solution

**Table 9-2**  
**Organic Performance Indicators**

Performance Indicator	Criteria	
	Volatiles by GC/MS	Semi-Volatiles by GC/MS
Instrument Tune and Calibration	Refer to OLM01.8	Refer to OLM01.8
Method (Laboratory) Blank	<CRQL (<5x CRQL for common lab contaminants) Daily plus 1/12 hours	<CRQL Daily plus 1/20 samples per extraction batch per matrix
Instrument Blank	N/A	N/A
Laboratory Duplicate	See Matrix Spike/ Duplicate	See Matrix Spike/ Duplicate
Surrogate	N/A	Every Sample Refer to OLM01.8 for % Recovery
Matrix Spike	Daily per run per matrix Refer to OLM01.8 for % Recovery	Daily per extraction plus 1/20 per matrix Refer to OLM01.8 for % Recovery
Matrix Spike Duplicate	Daily per run per matrix Refer to OLM01.8 for % Recovery and % RPD	Daily per extraction plus 1/20 per matrix Refer to OLM01.8 for % Recovery and % RPD
Internal Standard Area	Each sample 50 - 200% of amount in calibration standard	Each sample 50 - 200% of amount in calibration standard
Check Standard	SPCC and CCC per OLM01.8 requirements	SPCC and CCC per OLM01.8 requirements



## **SECTION 10**

### **DATA REDUCTION, VALIDATION AND REPORTING**

#### **10.1 FIELD MEASUREMENTS**

The field logbook will record raw data from field measurements and sample collection activities. No data reduction will take place in the field, and data validation will consist of checking for transcription errors and looking at field logs to ensure that instrument calibrations are done in accordance with appropriate SOPs.

#### **10.2 LABORATORY SERVICES**

##### **10.2.1 Data Reduction**

Data reduction is performed by the individual analysts and consists of calculating concentrations in samples from the raw data obtained from the measuring instruments. The laboratory will follow procedures specified in OLM01.8 and ILM02.1. The complexity of the data reduction will be dependent on the specific analytical method and the number of discrete operations (e.g., extractions, dilutions, and concentrations) involved in obtaining a sample that can be measured. The analyst will reduce or calculate all raw data into the final reportable values or enter all necessary raw data into LIMS in order for the database system to calculate the final reportable values. Copies of all raw data and the calculations used to generate the final results, such as hard-bound laboratory notebooks, strip-charts, chromatograms, Lotus spreadsheets, and LIMS record files, will be retained on file to allow reconstruction of the data reduction process at a later date.

For data reporting, rounding will not be performed until after the final result is obtained to minimize rounding errors, and results will not normally be expressed in more than two (2) or three (3) significant figures. All results will be reported with the proper measurement units (e.g., mg/L,  $\mu\text{g/kg}$ , etc.). The SOPs in Appendix E present the formulas to be used in determining the concentration of contaminants in samples.

### Data Reporting

Reports will contain final results (uncorrected for blanks and recoveries), blank and recovery results, methods of analysis, levels of detection, surrogate recovery data, and method blank data. In addition, special analytical problems, and/or any modifications of referenced methods will be noted. The number of significant figures reported will be consistent with the limits of uncertainty inherent in the analytical method. Consequently, most analytical results will be reported to no more than two (2) or three (3) significant figures. Data are normally reported in units commonly used for the analyses performed. Concentrations in liquids are expressed in terms of weight per unit volume (e.g., milligrams per liter [mg/L]). Concentrations in solid or semi-solid matrices are expressed in terms of weight per unit weight of sample (e.g., micrograms per gram [ $\mu\text{g/g}$ ]). WESTON-Gulf Coast Laboratories, Inc. uses the standard U.S. EPA data qualifiers in their laboratory reports (e.g., U, J)

Reported detection limits will account for all appropriate concentration, dilution, and/or extraction factors, unless otherwise specified. The laboratory will provide a full CLP data package for this project. The laboratory will provide copies of original data.

The final data report that will be provided by WESTON-Gulf Coast Laboratories, Inc. will be compiled as follows:

#### Inorganic Data Report

1. Cover letter with Laboratory Manager/Project Manager sign-off
2. Data Qualifiers
3. Chain of Custody
4. Lab chronicle describing: Client ID/Analysis, RFW #, Matrix, Prep #, Collection Date, Extr/Prep Date, Analysis Date, and Section Manager sign-off
5. Case Narrative
6. Client Data Report
7. Quality control summary reports: Method Blank Data Report, Precision Data Report, Accuracy Data Report, and Laboratory Control Standards Report (LCS)

The final data report will be given to the WESTON Project Managers, the WESTON Project Director, and the Techalloy Project Director. It will be available to the U.S. EPA upon request.

### 10.2.3 Data Validation

Data validation will be performed by trained WESTON personnel external to WESTON-Gulf Coast Laboratories. Validation will be accomplished by comparing the contents of the data packages and QA/QC results to the requirements contained in the method SOPs. The validation procedures will be based on the following U.S. EPA Region V validation protocol:

- Laboratory Data Validation Functional Guidelines for Evaluating Organic Analyses - U.S. EPA, February 1988.
- Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses - U.S. EPA, July 1988.

CLP data validation methods will be used even for non-CLP parameters, such as tin and TSS.



provided with the acceptable result for the unknown sample until after the experimental results are reported; however, it is known that the sample is a performance test. In a double-blind performance test, the analyst not only has no knowledge of the acceptable result, but the sample is disguised in such a manner as to maintain its anonymity as a performance test sample.

Systems audits and surveillance evaluate the operational details of the QA program. An audit provides a systematic procedure to check the implementation of a specific QA requirement, such as the tracking of samples or chain-of-custody procedures. Audits will be conducted by persons other than those who performed or directly supervised the work being inspected. A surveillance consists of inspection or monitoring of a specific targeted area for compliance to requirements, such as an evaluation of a single analytical method to ensure conformance with the written SOP.

The laboratory audit protocols outlined in this section pertain to WESTON-Gulf Coast Laboratories, Inc. If any additional or alternative laboratories are assigned to perform analyses for the Techalloy RFI via this QAPP, any changes to the audit procedures will be provided to the U.S. EPA for review and approval in a QAPP addendum.

Informal laboratory audits are conducted to verify that stated corrective actions are implemented. Alternative forms of laboratory audits are conducted daily based on a secondary review system that is implemented throughout the laboratory. Secondary reviews include laboratory notebooks, Lotus spreadsheets, organic packages, etc. This type of audit system allows the laboratory to achieve the highest degree of accuracy in documentation and data reporting prior to reporting data to the client.

### External Audits

The QA Manager of the WESTON-Gulf Coast Laboratories, Inc. is responsible for scheduling and coordinating all external audits. External performance and system audits of the laboratory are the responsibility of the U.S. EPA Region V Central Regional Laboratory.

## SECTION 13

### SPECIFIC ROUTINE PROCEDURES TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS

#### 13.1 FIELD MEASUREMENTS

Field data will be assessed by the field team leader or his designee. The field team leader or his designee will review the field results for compliance with the established QC criteria that are specified in the QAPP. Accuracy of the field measurements will be assessed using daily instrument calibration and QC checks with standard solutions.

Precision will be assessed on the basis of analysis of duplicates. Data completeness will be calculated using Equation 13-1.

$$\text{Completeness} = \frac{\text{Valid Data obtained}}{\text{Total Data Planned}} \times 100 \quad \text{Equation 13-1}$$

#### 13.2 LABORATORY DATA

Laboratory results will be assessed for compliance with required precision, accuracy, completeness and sensitivity as discussed in the following sections.

##### 13.2.1 Precision

Precision of laboratory analysis will be assessed by comparing the analytical results between MS/MSD for organic analysis and laboratory duplicates for inorganic analysis. The relative

percent difference (RPD) will be calculated for each pair of duplicate analysis using Equation 13-2.

$$\%RPD = \frac{S - D}{(S + D)/2} \times 100 \quad \text{Equation 13-2}$$

Where: S = First sample value (original or MS value)  
D = Second sample value (duplicate or MSD value)

### 13.2.2 Accuracy

Accuracy of laboratory results will be assessed for compliance with the established QC criteria that are described in Section 4 of the QAPP, using the analytical results of method blanks, reagent/preparation blank, and matrix spike/matrix spike duplicate samples. The percent recovery (%R) of matrix spike samples will be calculated using Equation 13-3.

$$\%R = \frac{A - B}{C} \times 100 \quad \text{Equation 13-3}$$

Where:

- A = The analyte concentration determined experimentally from the spike sample;
- B = The background level determined by a separate analysis of the unspiked sample and;
- C = The amount of the spike added.



**PROCEDURE FOR COMPLETING THE WESTON-ANALYTICS  
CHAIN-OF-CUSTODY FORM**

The following information will be provided on the WESTON-Analytics chain-of-custody form.

Area 1

This section contains general project information.

Client: Techalloy RFI

Work Order No.: (A WESTON internal tracking number will be placed here.)

Project Contact/

Phone Number: Carlos Serna (708) 918-4002

Area 2

Sampling container packaging and preservation information is presented in this section.

Example: #/Type of Container: 1-glass; 2-P.E. (polyethylene)

Volume: 80 mL; 2 liters

Preservatives: HNO<sub>3</sub> (nitric acid), HCL (Hydrochloric Acid), NA (not applicable).

Area 3

Sample identification numbers are presented in this section.

Example: TC1-CP02-SBS-MS/MSD

Area 4

Quality control information is identified in this section. If the sample is assigned for MS/MSD analyses or spike/duplicate analyses, these columns are checked (✓ or X).

Area 5

The sample matrix is identified in this column.

Example: soil, water

**Table 2-2**  
**Summary of Sampling Effort**  
**Techalloy Company, Inc.**  
**Union, Illinois**

Sample Matrix	Field Parameters	Laboratory Parameters	Investigative			Field Duplicate			Field Blank			Matrix Spike/ Matrix Spike Duplicate <sup>1</sup>			Matrix Total <sup>2</sup>
			No.	Freq.	Total	No.	Freq.	Total	No.	Freq.	Total	No.	Freq.	Total	
Phase I															
SWMU Soil Borings															
Wire Slag Disposal Area	Description and classification	VOCs	4	1	4	1	1	1	—	—	—	1	1	1	5
		Metals	4	1	4	1	1	1	—	—	—	—	—	—	5
BG-5 Oil Drum Storage Area	Description and classification	VOCs	10	1	10	1	1	1	—	—	—	1	1	1	11
		Metals	10	1	10	1	1	1	—	—	—	—	—	—	11
Spent Acid Holding Pond	Description and classification	VOCs	27	1	27	3	1	3	—	—	—	2	1	2	30
		Metals	27	1	27	3	1	3	—	—	—	—	—	—	30
Plating Wastewater Disposal Area	Description and classification	VOCs	10	1	10	1	1	1	—	—	—	1	1	1	11
		Metals	10	1	10	1	1	1	—	—	—	—	—	—	11
		CN	10	1	10	1	1	1	—	—	—	—	—	—	11
Concrete Evaporation Pad	Description and classification	VOCs	18	1	18	2	1	2	—	—	—	1	1	1	20
		Metals	18	1	18	2	1	2	—	—	—	—	—	—	20
Background soils	Description and classification	Metals	12	1	12	2	1	2	—	—	—	—	—	—	7
Total			81	5	81	10	1	10	—	—	—	7	1	7	91

Table 2-2

Summary of Sampling Effort  
 Techalloy Company, Inc.  
 Union, Illinois  
 (Continued)

Sample Matrix	Field Parameters	Laboratory Parameters	Investigative			Field Duplicate			Field Blank			Matrix Spike/ Matrix Spike Duplicate <sup>1</sup>			Matrix Total <sup>2</sup>
			No.	Freq.	Total	No.	Freq.	Total	No.	Freq.	Total	No.	Freq.	Total	
SWMU Groundwater															
Wire slag disposal area	pH, temperature, specific conductance	VOCs	1	1	1										
		SVOCs	1	1	1										
		Metals-filtered	1	1	1										
		Metals-unfiltered	1	1	1										
		TSS	1	1	1										
BG-5 oil drums	pH, temperature, specific conductance	VOCs	1	1	1										
		SVOCs	1	1	1										
		Metals-filtered	1	1	1										
		Metals-unfiltered	1	1	1										
		TSS	1	1	1										
Spent acid holding pond	pH, temperature, specific conductance	VOCs	5	1	5										
		SVOCs	1	1	1										
		Metals-filtered	5	1	5										
		Metals-unfiltered	5	1	5										
		TSS	5	1	5										



### **BG-5 Oil Drums**

Five soil borings (BG-01 through BG-05) will be advanced near the former BG-5 oil drum staging area to characterize this potential source area (Figure 2-2). Boring BG-03 will be located at the center of the former drum staging location. The other four borings will be located 30 feet to the north, south, east, and west of the unit to determine the lateral extent of potential constituent migration. This placement of the borings encompasses the drum staging area and the area north of the facility structure and south of the spent acid holding pond sampling area. Vadose zone soil samples will be collected for analysis from 1 to 2 feet below the native soil surface, to avoid sampling any surficial fill material (if present). Vadose zone soil samples will also be collected at 5 to 6 feet bgs, above the capillary fringe of the water table. Boring BG-03, located at the center of the BG-5 area will be advanced below the water table to allow for collection of a groundwater sample to determine if constituents related to the oil drums have migrated vertically to the groundwater pathway (Table 2-2).

### **Spent Acid Holding Pond**

Fifteen soil borings (HP-01 through HP-15) will be advanced in and around the former holding pond to characterize the magnitude and lateral and vertical extent of potential constituent migration from this potential source area (Figure 2-2). Borings HP-07 through HP-09 will be advanced through the bottom of the holding pond to characterize the constituents that may have infiltrated the basal soil. The base of the pond is approximately 4 feet bgs and approximately 3 feet above the capillary fringe of the water table. One vadose zone soil sample will be collected from within this 3-foot interval from each of the three borings for analysis. This sampling depth is approximately 5 to 6 feet bgs. These three samples will be collected with a small diameter manually-driven bucket auger.

The remaining 12 borings will be located around the perimeter of the holding pond. The boring locations consist of three east-west rows of five borings. The boring locations are spaced 100 feet east-west and 70 feet north and 50 feet south of the long axis of the holding pond (Figure 2-2). This placement of the borings encompasses the perimeter of the holding pond. The borings extend from immediately north of the sampling areas that encompass

sample collection with minimal soil cuttings generated. The Geoprobe will reduce the time and expense required to collect the soil and groundwater samples specified in this FSP. It will also eliminate the time and expense required to characterize and dispose of potentially contaminated soil cuttings.

The Geoprobe soil and groundwater sampling procedures are described in Section 3. The following is a description of the drilling locations and sampling activities to be performed at each SWMU area.

#### **Wire Slag Disposal Area**

Two soil borings (WS-01 and WS-02) will be advanced near the dumpster containing the wire slag (Figure 2-2). Because the surrounding area is covered with concrete, impact to the underlying soils is considered unlikely. Therefore, only two soil borings will be advanced to characterize this potential source area. The two borings will be located immediately west and east of the dumpster, respectively, to determine if soils have been impacted. The borings will be advanced through the concrete surface and two vadose zone soil samples will be collected for analysis from each boring (Table 2-2). Soil samples will be collected at 1 to 2 feet below the soil/backfill interface and at 5 to 6 feet bgs, above the capillary fringe of the water table.

Boring WS-01, located downgradient and west of the dumpster will be advanced below the water table to allow for collection of a groundwater sample. The groundwater analysis (Table 2-2) will determine if constituents related to this SWMU have migrated vertically to the groundwater pathway.



Borings (CP-01, CP-02, CP-05, CP-06, CP-08, CP-09, and CP-10) will be advanced around the perimeter of the evaporation pad. These borings are spaced 50 feet north-south and east-west from boring CP-04 (located within the evaporation pad) to determine the lateral and vertical extent of potential constituent migration. Boring CP-07 is located immediately east of the pad and boring CP-03 is located immediately north of the pad to determine the impact to the underlying soils immediately adjacent to the pad.

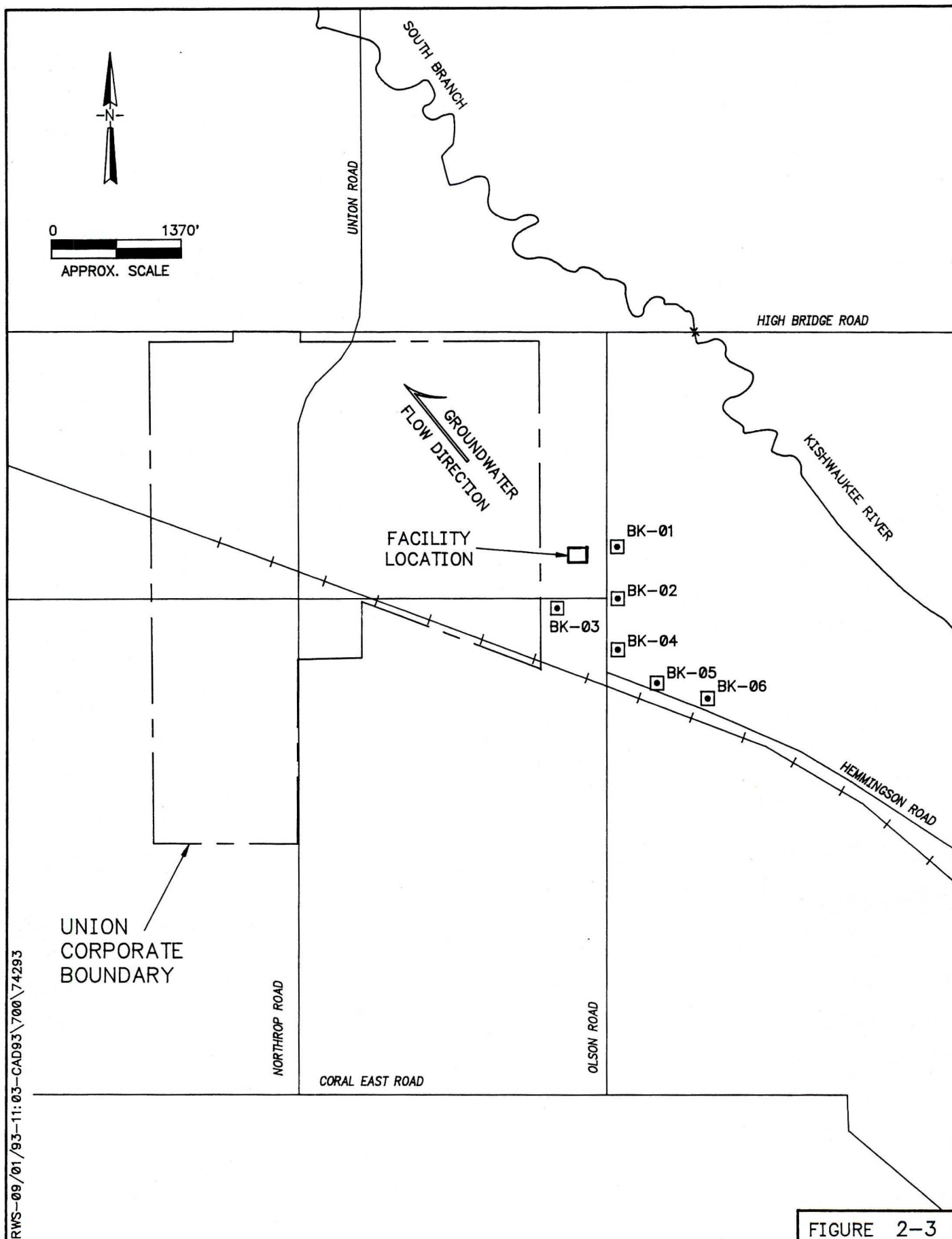
As indicated earlier, due to the proximity of the plating wastewater disposal area to the concrete evaporation pad, vadose zone soil samples collected from boring CP-03 will be analyzed for the plating wastewater parameters (VOCs, selected Appendix IX inorganics and cyanide). This analysis will also encompass the evaporation pad parameters (VOCs and selected Appendix IX metals) and will provide data relevant to both SWMUs. This placement of the borings surrounds the evaporation pad on the south, east and north sides of the pad. This sampling area extends west to the plating wastewater sampling area, north to the holding pond sampling area, and east and south to encompass the area potentially impacted by the evaporation pad.

Boring CP-03, located on the north side of the pad, will be advanced below the water table to allow for collection of a shallow groundwater sample. The sample analysis will determine the magnitude of the impact to the groundwater pathway at this location. The boring will then be advanced to the base of the aquifer at 35 feet bgs for the collection of a deep groundwater sample. The deep sample analysis will determine the impact to the deep portion of the groundwater pathway at this location. The groundwater analysis from boring CP-03 is included with the total number of concrete evaporation pad groundwater analyses on Table 2-2.

### **Background**

Six soil borings (BK-01 through BK-06) will be advanced at locations upgradient (southeast) of the Techalloy facility (Figure 2-3). Each boring will be sampled at 1 to 2 feet and 5 to 6 feet bgs, similar depths as those borings on site. Each boring will also be advanced below the water table for collection of a shallow groundwater sample. The soil and groundwater samples will be analyzed according to the scheme outlined in Table 2-2. The purpose of





Three Hawthorn Parkway  
Vernon Hills, Illinois  
60061

PHASE I BACKGROUND  
SOIL BORING LOCATIONS  
TECHALLOY COMPANY, INC.  
Union, Illinois

RFI will consist of the previously-described groundwater sampling at each SWMU and the measuring of water levels and sampling of the existing monitoring wells.

The recording of water table elevations in on- and off-site monitoring wells will be performed to confirm the current groundwater flow direction. Two rounds of water levels will be recorded during the RFI.

The 13 existing monitoring wells will be sampled for VOCs and metals analysis to establish the current distribution of constituents in on- and off-site groundwater. Six groundwater samples will also be collected from soil borings advanced below the water table at six background locations upgradient of the Techalloy facility. The upgradient groundwater analytical results will be compared statistically to the on-site and downgradient results to determine the significance of those detections. This information, in addition to the previous plume characterization work, will characterize the current groundwater pathway scenario.

### **2.3 PHASE II INVESTIGATION ACTIVITIES**

The Phase II source characterization activities are contingent on the results of the Phase I analyses. If the Phase I results do not meet the objectives of the RFI, additional sample locations will be identified and sampled under a Phase II effort. For example, if the horizontal and vertical extent of constituent migration is not fully identified around a particular SWMU, additional borings will be advanced and sampled in a manner consistent with the Phase I methods to fully characterize the area.

The Phase II pathway characterization activities are contingent on the analytical results of the Phase I activities. If determined to be necessary, additional groundwater monitoring wells will be installed and sampled to characterize constituent concentrations and/or flow characteristics in specific areas.

## **2.4 POTENTIAL RECEPTORS**

The potential receptors of constituents migrating downgradient of the facility with groundwater have been identified. Those residences with private water wells, located in proximity to the groundwater plume and exhibiting VOCs in previous chemical analyses, have been designated for future testing. In compliance with the consent order, the details of the potential receptor investigative activities have been presented to U.S. EPA in the "Private Well Sampling Plan," dated February 1993.



## SECTION 3

### FIELD INVESTIGATION PROTOCOLS

The following subsections describe the procedures to be used during the RFI at the Techalloy facility. Specifically, these subsections contain protocols for soil borings, sampling of soil and groundwater, sample collection, field testing, and monitoring well installation.

#### 3.1 SOIL BORING SAMPLING

Soil samples will be collected for chemical analysis from two distinct depths in each soil boring except borings HP-07 through HP-09, which will be sampled at one depth. Samples will be analyzed according to the analytical scheme outlined in Table 2-2. The following steps will be used for the advancement and sampling of all soil borings:

- A hydraulic push/pull mechanism (Geoprobe) mounted on a four-wheel-drive truck will be utilized to advance all soil and groundwater sampling borings.
- The working end of the Geoprobe and all boring equipment, tools, and materials will be decontaminated prior to advancing a boring at each location in accordance with the protocols presented in Table 3-1. Provisions will be made to prevent equipment, tools, and materials from coming into contact with surface soils.
- The Geoprobe will advance a decontaminated 1-inch diameter by 2-foot-long bore sampler with a retractable drive point to the upper limit of the first desired sampling interval. The sampling equipment will be decontaminated in accordance with the protocols in Table 3-2.
- If the sampler meets with resistance and will not push to a sufficient depth, a hydraulic hammer will be used to advance the sampler.
- At the desired sample depth, the retractable drive point is released and the sampler advanced through the desired interval. Soil from this interval is collected into the sampling sleeve.

registered Professional Land Surveyor will determine the elevations at the conclusion of the field investigative activities.

- Water table elevations will be plotted on an area map and water level contours will be constructed to determine horizontal flow conditions.
- Water table elevations measured in shallow and deep wells will be compared to establish the vertical component of groundwater flow.
- Groundwater depth and flow direction will be determined on and off site.

An SOP for water level measurements has been included in Appendix B.

### **3.4 GROUNDWATER MONITORING WELL INSTALLATION**

If the results of the Phase I RFI activities determine that additional groundwater monitoring points are required, additional groundwater monitoring wells will be installed under Phase II of the RFI. The additional shallow groundwater monitoring wells will be installed to screen the water table and uppermost portion of the aquifer. The shallow well screens will be installed from 2 feet above to 8 feet below the water table. If additional deep monitoring wells are required to monitor water quality at the base of the aquifer, the well screens will be installed to screen the bottom 10 feet of the aquifer.

The following steps will be used during the installation of all groundwater monitoring wells:

- The borings for shallow monitoring wells will be advanced to their desired depths below the water tables, utilizing a high-torque drill rig and 4.25-inch hollow-stem augers (HSAs).
- The borings for deep monitoring wells will be advanced to their desired depths utilizing mud rotary drilling techniques.
- The working end of the drill rig and all drilling equipment, tools, and materials will be decontaminated prior to drilling at each location in accordance with protocol presented in Table 3-1. Provisions will be made to keep equipment, tools, and materials from coming into contact with surface soils.



- Samples for the additional analyses will then be transferred into the appropriate sample containers. The sequence for filling the remaining sample containers is as follows: semivolatiles, selected Appendix IX inorganics, cyanide, then total suspended solids (TSS).
- All organic samples, TSS, and the inorganic cyanide aliquot will be submitted to the laboratory unfiltered. Two total metals samples will be collected at each location where groundwater samples will be collected for RCRA metal analysis. One metals sample will be submitted to the laboratory unfiltered, the second sample will be filtered in the field. The filtered water sample for metals analysis will be pumped from a sample container, through an in-line 0.45-micron filter via polyethylene tubing using a peristaltic pump. The filtered groundwater sample will be collected in an unused laboratory-prepared sample container. Sample preservation of the filtered sample will be performed after filtration.

Samples will be analyzed according to the analytical scheme outlined in Table 2-2.

### **3.6 SPLIT-SPOON SAMPLING PROCEDURES**

Following removal from the borehole, the split-spoon sampler will be opened on a clean surface (e.g., polyethylene sheeting). Sample collection will commence immediately upon opening the split-spoon in order to minimize the loss of VOCs. The VOC sample will be collected first as a grab sample. Sample material from several places along the core at the depth of interest will be removed using a decontaminated stainless steel spatula, spoon, or scoop, and placed as quickly as possible into the assigned VOC sample containers. No mixing or compositing will be performed on the VOC sample material, thereby limiting the loss of volatile organic compounds from the sample. The VOC sample container will be filled completely, packing the soil in the container as tightly as possible. The remainder of the sample material will be homogenized and placed in the appropriate remaining sample containers. These samples will be considered composite samples. The homogenization procedure is presented in Subsection 3.7.



## **SECTION 5**

### **SAMPLE HANDLING**

#### **5.1 SAMPLE CONTAINERS AND SAMPLE PRESERVATION**

All soil and groundwater samples are expected to be low hazard levels. Table 5-1 lists the required sample containers, sample volumes, sample preservation requirements, and holding times associated with all parameters and media applicable to the Techalloy RFI sampling activities.

#### **5.2 SAMPLE PACKAGING AND SHIPMENT**

All samples shipped from the Techalloy facility must be shipped in accordance with U.S. Department of Transportation (DOT) regulations and must comply with Dangerous Goods Regulation [International Air Transport Association (IATA), (1993)] if shipped by air transport.

Following sample collection, the exteriors of all sample containers will be wiped clean with a moist cloth. The filled sample containers will not be sprayed with water during decontamination because this water could contact the sample if the container is not tightly sealed. In preparation for shipment to the WESTON-Gulf Coast Laboratories, all samples will be packaged in accordance with the following procedures:

- Each sample container will be checked to ensure that the container lid is securely tightened.
- Each sample container will be checked to ensure that the sample label has been securely affixed to the container and completely/correctly filled out with the appropriate sample I.D. number, sample, date, and analytical parameters as a minimum requirement.

**Table 5-1**

**Required Sample Containers, Volumes, Preservation, and Holding Times  
 Techalloy Company, Inc.  
 Union, Illinois**

Material Type	Analysis	Number of Containers	Required Sample Volume	Sample Container Type	Sample Preservation	Sample Holding Time <sup>1</sup>
Soil	VOCs <sup>1,2</sup>	1	4 oz.	4 oz. wide-mouth glass jar with Teflon® - lined cap	Cool to 4°C	14 days for extraction and analysis
	Metals and cyanide	1	8 oz.	8-oz. wide-mouth glass jar	Cool to 4°C	6 months (28 days for mercury) (14 days for cyanide)
Groundwater	VOCs <sup>2,3</sup>	2	80 mL	40-mL glass vials with Teflon® - lined caps	HCl to pH <2 and cool to 4°C	14 days for analysis
	Metals	1	1 Liter	1 Liter polyethylene bottle.	Nitric acid to pH <2 and cool to 4°C	6 months for analysis
	SVOCs	2	160 oz.	80 oz. glass amber bottles with Teflon® - lined caps	Cool to 4°C	7 days for extraction analysis within 40 days
	TSS	1	1 liter	1 liter polyethylene bottle	Cool 4°C	7 days for analysis

<sup>1</sup>All holding times are calculated from the date of sample collection.

<sup>2</sup>Duplicate analysis requires the sample to be collected at double the volume specified.

<sup>3</sup>MS/MSD for organics and spike/duplicate for metals and cyanide analyses require the sample to be collected at double the volume specified.

Note: One trip blank will accompany each aqueous VOC shipment container. Trip blanks will consist of two 40 mL vials.

# STANDARD OPERATING PROCEDURE

## WATERLEVEL MEASUREMENT

### 1.0 PURPOSE

This procedure describes the method for determining the depth-to-water in a monitoring well or piezometer.

### 2.0 DISCUSSION

Generally, water level measurements from piezometers or monitoring wells are used to construct potentiometric surface maps. Therefore, all water level measurements at a given site should be collected within a 24-hour period. Under the following conditions, all measurements must be taken within a shorter interval.

- A range of observed changes between wells that is too large to be indicative of natural gradient groundwater.
- Drastic atmospheric pressure changes.
- Tidally influenced aquifers.
- Aquifers affected by river stage, impoundments, or unlined ditches.
- Aquifers stressed by intermittent pumping of production wells.
- Aquifers being actively recharged because of a precipitation event or induced infiltration.

The device used to measure water levels should be adequate to attain an accuracy of 0.01 feet. Generally acceptable devices are listed below.

- Chalked steel tape.
- Fiberglass tape with a popper.
- An electric sounder.



There should be a survey mark on the piezometer or well casing as a reference measuring point. If there is not a survey mark, place one on the casing. The mark should be permanent.

Allow water levels in piezometers and monitoring wells to stabilize for a minimum of 24 hours after well construction and development before measurements are taken. Recovery may take longer in wells completed in tight formations.

### **3.0 PROCEDURES**

- A. Obtain a logbook from the site manager or other project-designed individual.
- B. Locate monitoring wells or piezometers to be measured and the appropriate decontamination area.
- C. Decontaminate all sampling equipment before taking the first measurement and between measurement intervals.
- D. When taking a number of water level measurements, it is preferable to start at those wells that are the least contaminated and proceed to those wells that are the most contaminated.
- E. Whenever a water level is to be measured, enter a description of the measuring location into the logbook.
- F. Remove locking well cap. Note the location, time of day, weather conditions, and date in the logbook.
- G. If required by site-specific conditions, monitor the headspace of the well with a photoionization detector or a flame ionization detector to determine the presence of volatile organic compounds and record the measurements in the logbook.
- H. Lower the measuring device into the well until the water surface is encountered.
- I. Measure the total depth of the well and the distance (in feet, tenths of feet, or hundredths of feet) from the water surface to the reference measuring point on the well casing. Record the well depth and distance to water in the logbook.
- J. If the total depth of the well has increased, and/or if the well appears to have heaved, a re-survey of the vertical elevation must be performed. If the total well depth has decreased, the well screen may have silted in, and redeveloping may be needed. Notify the Site Manager if either of these situations has occurred.

- K. Measure depth-to-water at least twice or until results are reproduced and record the measurement in the logbook.
- L. Remove all downhole equipment. Replace the well casing cap and locking steel caps.
- M. Decontaminate all downhole equipment and store for transport to the next measuring location.
- N. Note any physical changes (like erosion or cracks) in the protective concrete pad or variation in the total depth of the well in the logbook. Check the operational condition of the padlock.

#### Area 6

The date of sample collection will be presented in this section for each sample identified in Area 3.

Example: 4/16/93

#### Area 7

The time of sample collection for each sample identified in Area 3 will be specified in this column in military time.

Example: 1420 presenting 2:20 P.M.

#### Area 8

The required analyses/parameters are specified via a check mark (✓ or X) in the appropriate box.

#### Area 9

Each required analyses/parameter being requested is identified in this section. Additional parameters can be added in the blank boxes.

#### Area 10

Any specific instructions or special information associated with the samples identified in Area 3 will be noted by the Field Sample Manager in this section.

Example:     -     If the sample exhibited unusual characteristics (e.g., odor, elevated PID readings).

              -     If the sample requires special handling at the laboratory.

#### Areas 11, 12, 13, 14

In these areas, the field person relinquishing the samples to the courier will acknowledge release of the samples. His/her signature will be placed in Area 11. Area 12 will remain blank. The relinquisher will state the date and time the samples left his/her custody in Areas 13 and 14, respectively.

#### Areas 15, 16, 17, 18

Upon receipt at the laboratory, the sample custodian logging in the samples will acknowledge receipt of the samples in these sections. Area 15 is left blank. The custodian's signature is placed in Area 16, and the time and date at which he/she acknowledges receipt of the samples will be stated in Areas 17 and 18, respectively.



Area 19

Following receipt of the sample shipment container(s), the laboratory sample custodian will check the samples received and complete this section based on his/her review.

**WESTON Analytics Use Only**

## Custody Transfer Record/Lab Work Request

**WESTON**

Page of

[illegible]

RFW 21-21-001/A-7/91

L372

L373

L375

L377

L378

Rel#

Cooler #

381 596

FIGURE



Three Hawthorn Parkway  
Vernon Hills, Illinois  
60061

WESTON - ANALYTIC  
CHAIN-OF-CUSTODY FORM  
TECHALLOY COMPANY, INC.  
Union, Illinois

Table 2-2

Summary of Sampling Effort  
 Techalloy Company, Inc.  
 Union, Illinois  
 (Continued)

Sample Matrix	Field Parameters	Laboratory Parameters	Investigative			Field Duplicate			Field Blank			Matrix Spike/ Matrix Spike Duplicate <sup>1</sup>			Matrix Total <sup>2</sup>
			No.	Freq.	Total	No.	Freq.	Total	No.	Freq.	Total	No.	Freq.	Total	
Plating wastewater disposal area <sup>3</sup>	pH, temperature, specific conductance	VOCs	1	1	1										
		SVOCs	1	1	1										
		Metals-filtered	1	1	1										
		Metals-unfiltered	1	1	1										
		TSS	1	1	1										
Concrete evaporation pad	pH, temperature, specific conductance	VOCs	2	1	2										
		SVOCs	1	1	1										
		Metals-filtered	2	1	2										
		Metals-unfiltered	2	1	2										
		TSS	2	1	2										
Total			10	1	10	1	1	1	1	1	1	1	1	1	12
Groundwater															
Existing monitoring wells <sup>4</sup>	pH, temperature, specific conductance	VOCs	13	1	13	2	1	2	2	1	2	1	1	1	17
		Metals-filtered	13	1	13	2	1	2	2	1	2	—	—	—	17
		Metals-unfiltered	13	1	13	2	1	2	2	1	2	—	—	—	17
		TSS	13	1	13	2	1	2	2	1	2	—	—	—	17



Table 2-2

Summary of Sampling Effort  
 Techalloy Company, Inc.  
 Union, Illinois  
 (Continued)

Sample Matrix	Field Parameters	Laboratory Parameters	Investigative			Field Duplicate			Field Blank			Matrix Spike/ Matrix Spike Duplicate <sup>1</sup>			Matrix Total <sup>2</sup>
			No.	Freq.	Total	No.	Freq.	Total	No.	Freq.	Total	No.	Freq.	Total	
Groundwater (Cont.)															
Background groundwater	pH, temperature, specific conductance	VOCs	6	1	6	1	1	1	1	1	1	1	1	1	8
		SVOCs	6	1	6	1	1	1	1	1	1	1	1	1	8
		Metals-filtered	6	1	6	1	1	1	1	1	1	---	---	---	8
		Metals-unfiltered	6	1	6	1	1	1	1	1	1	---	---	---	8
		TSS	6	1	6	1	1	1	1	1	1	---	---	---	8
Total			19	1	19	3	1	3	3	1	3	2	1	2	25
Phase II															
Soil and Groundwater															
New monitoring wells	pH, temperature, specific conductance	Dependent on Phase I results													
Matrix, number, and analyses dependent on Phase I results															

Notes: Matrix totals do not include trip blank samples; VOC trip blank samples will be shipped and analyzed at a frequency of one per shipping container of aqueous VOC samples. SVOCs include polyaromatic hydrocarbons (PAHs).

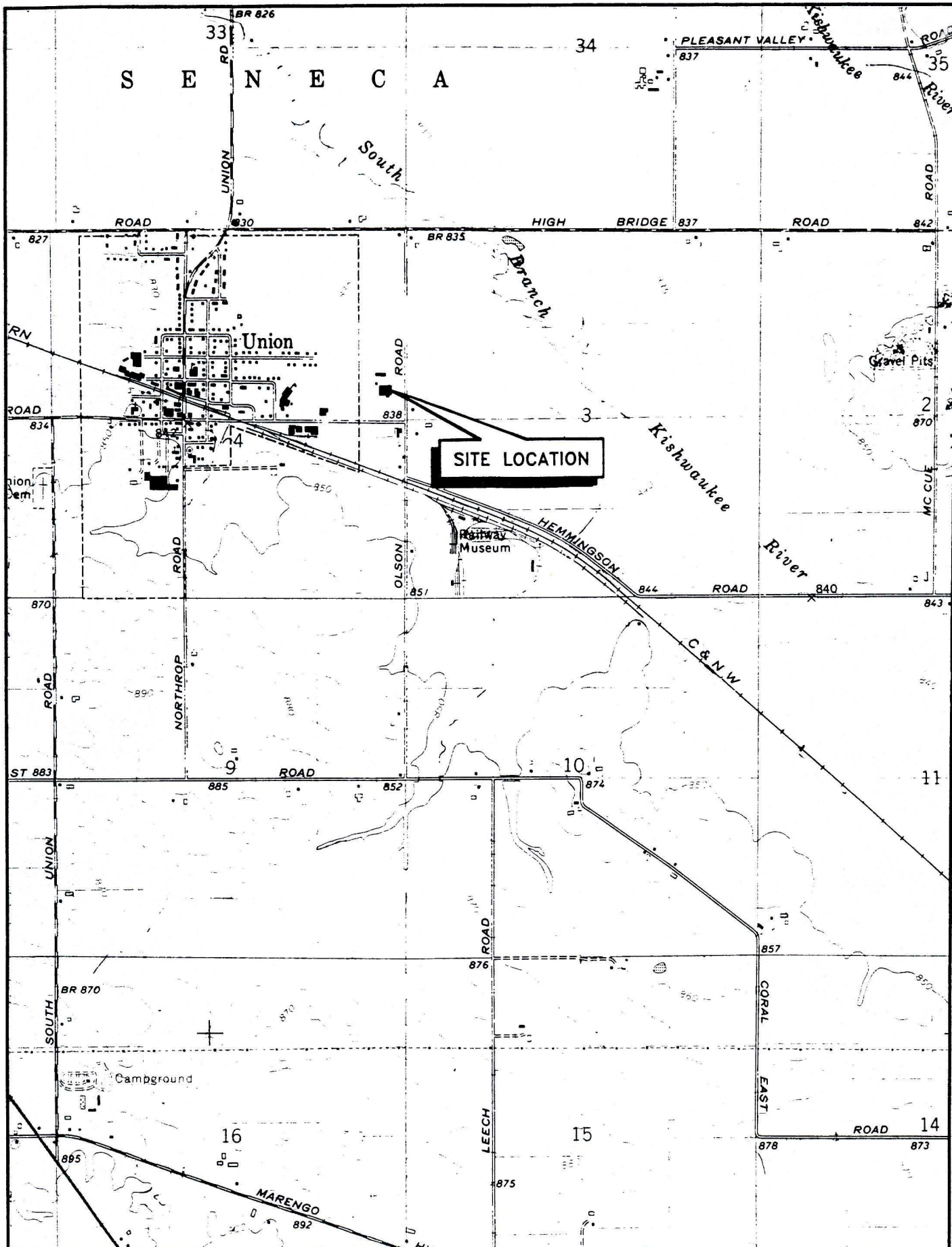
Two soil samples from boring CP-03 (Concrete Evaporation Pad area) will be analyzed for the plating wastewater disposal area parameters due to the proximity of the wastewater area. The analyses of these two samples are counted with the plating wastewater disposal area samples.

<sup>1</sup>MS/MSDs are not additional samples, but are instead investigative samples on which MS/MSD analyses are performed. MS/MSD analyses are for organic samples only and requires the sample to be collected at double the volume specified for aqueous media. Duplicate/spike analyses will be performed for inorganic samples.

<sup>2</sup>The matrix total does not include trip blank samples, MS/MSDs and duplicate/spike samples. One trip blank sample will be shipped with every shipment container of aqueous VOA samples.

<sup>3</sup>The groundwater sample will be collected from existing monitoring well MW-10.

<sup>4</sup>One groundwater sample will be collected from each of the 13 existing monitoring wells.



**WESTON**  
MANAGERS DESIGNERS/CONSULTANTS

Three Hawthorn Parkway  
Vernon Hills, Illinois  
60061

FIGURE  
1-1

SITE LOCATION  
TECHALLOY COMPANY, INC.  
Union, Illinois

REV.



In February 1991, WESTON initiated a Phase II investigation to further define the groundwater contaminant plume. Groundwater probe samples were collected downgradient from the Techalloy facility in order to delineate the lateral extent of contamination. These samples were analyzed on site for trichloroethane (TCA), trichloroethene (TCE) and tetrachloroethene (PCE).

In order to confirm the groundwater probe results and define the vertical extent of constituents, additional groundwater samples were collected through lead screen augers and sent to WESTON-Gulf Coast Laboratories in University Park, Illinois for analysis. The results of both sampling events are summarized on Figure 2-10 of the QAPP. The probe sample locations are designated WS, and the lead screen auger sample locations are designated TW.

### 1.3 SUMMARY OF PREVIOUS PRIVATE WELL SAMPLING

Techalloy completed four rounds of private well sampling from June 1990 to September 1991. Groundwater samples were collected from 17 private wells, with a different number and combination of wells sampled during each sampling event. The locations of the 17 wells are presented in Figure 1-2. Analytical results for the four rounds of sampling are summarized in Tables 1-1 through 1-4. Table 1-5 summarizes the sampling dates and analytical procedures associated with each round of sampling. Analytical results indicated that 6 of the 17 private wells had detectable levels of VOCs. TCE was the only VOC detected above the maximum contaminant level (MCL) of 5.0 µg/L, and was detected at the private well located at the northeast corner of **Non-responsive**. This well is located within the identified constituent plume.

The procedures used for collecting private well samples were consistent with the procedures presented in Section 2 of the PWSP.





ANALYTICS DIVISION  
**STANDARD PRACTICES  
MANUAL**  
COMPANY CONFIDENTIAL AND PROPRIETARY

OPERATING PRACTICE  
**Total and Volatile Suspended  
Solids**

Eff. Date: 03/19/93 Initiated By: QC Department Approved By: D. L. Harper *DLH* Authorized By: A. M. Henry SP No. 21-15G-160.2

RELEASED  
2019-007454  
July 27, 2020 - TJW

**INORGANIC ANALYSIS PROTOCOL  
Total and Volatile Suspended Solids  
(Non-Filterable Residue)**

**CONTROLLED DISTRIBUTION**

**COPY # :** *Uncontrolled*

**ISSUED TO :** *Techalloy QAPP*

Full Signature Approvals Are Kept on File  
with WESTON®'s Analytics Division  
QA Standard Practice Records

REVISION NUMBER: 01

1.0 **PURPOSE**

To determine the total non-filterable residue content in drinking, surface, and ground waters, domestic and industrial liquid wastes.

2.0 **REFERENCE**

This SOP was written using the following methods as references:

For Total Suspended Solids (TSS), EPA 600/4-79-020, Method 160.2 and Standard Methods, 17th Ed., Method 2540D.

For Volatile Suspended Solids (VSS), EPA 600/4-79-020, Method 160.4 and Standard Methods, 17th Ed., Method 2540E.

3.0 **METHOD SUMMARY**

3.1 Suspended solid matter in natural or man-made water bodies is of environmental importance because the presence of such material decreases the amount and depth of light penetration to the water body. The decrease in light penetration then decreases the potential for productivity of zooplankton and photoplankton of the water body, limiting the available food sources to higher vertebrates and invertebrates that are necessary to keep the ecosystem in balance.

3.2        Wastewater treatment plant operators use this information as a guide to pump performance within the plant and can often troubleshoot potential future problems. Final effluents are monitored to evaluate overall plant performance as well as to provide estimations of the suspended solids loading to the receiving stream.

3.3        Suspended solids material occurs naturally as silt, leaves, etc., and is caused by a variety of naturally occurring processes such as rain, decay, animal activities, etc. Litter and industrial effluents are sources of manmade suspended solids pollutants. Suspended solids materials are easily removed by filtration.

3.4        A sample may contain two types of solids, dissolved solids, and suspended solids. The suspended solids are those particles retained by a micro-fiber filter while the dissolved solids pass through the filter. The type of filter holder, the pore size, porosity, area and thickness of the filter and the physical nature, particle size and amount of material deposited on the filter are the principal factors affecting the trapping of suspended solids materials. The residue remaining on the filter paper is dried to a constant weight at 103-105°C.

If the TSS is less than the reporting limit, then VSS will not need to be determined.

4.0        **INTERFERENCES**

Exclude large floating particles or submerged agglomerates of non-homogeneous materials from the sample if they are not representative of the sample. Because excessive residue on the filter may form a water-entrapping crust, limit the sample size to that yielding no more than 200 mg of residue. For samples high in dissolved solids, thoroughly wash the filter to ensure removal of dissolved material. Prolonged filtration times resulting from filter clogging may produce high results owing to excessive solids caught on the clogged filter.

**5.0      SAMPLE COLLECTION, PRESERVATION AND HANDLING**

Holding time, preservation techniques and sample container may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client request. Listed below are the holding times, and the references which include container and preservation requirements for compliance with the Safe Drinking Water Act (SDWA) and the Clean Water Act (CWA).

<u>Regulation</u>	<u>Holding Time</u>	<u>Reference</u>
CWA NPDES	7 days	CFR 40 pt. 36.3
SDWA	7 days	EPA-570/9-82-002

All analyses are performed on unpreserved samples.

**6.0      INSTRUMENT AND EQUIPMENT**

- Analytical Balance - capable of weighing to 0.1 mg
- 103-105°C oven
- desiccator
- vacuum
- 100 mL graduated cylinder
- suction flask (side armed)
- crucible holder
- gooch crucibles, 40 mL
- Whatman 934-AH glass fiber filters
- 10 mL class A pipettes

**7.0      PREVENTATIVE MAINTENANCE**

- 7.1 Calibration check of balance, daily
- 7.2 Calibration check of 103-105°C oven, daily
- 7.3 Desiccant is inspected daily by analyst



## 8.0 STANDARDS AND REAGENTS

All standards and reagents are prepared with Type II Deionized Water unless otherwise stated.

### 8.1 Stock Solution I; 200 mg/L

Weigh out 200 mg diatomaceous earth (DE) to the nearest 1 mg and add it to 500 ml DI water in a 1.0 L volumetric flask. Dilute to volume with DI water and mix thoroughly.

- Life of QC Solution: one day
- Storage Requirements: none

### 8.2 Stock Solution II; 25 mg/L

Prepare as above using 25 mg DE rather than 200 mg.

- Life of QC Solution: one day
- Storage Requirements: none

## 9.0 PROCEDURE

### 9.1 Reporting Limit.....5 mg/L (based on a 100 mL minimum sample)

### 9.2 Sample Size

Working sample size varies, generally 100 ml is sufficient, but it is acceptable to increase or decrease the volume to obtain a range of at least 1.0 mg of residue, not to exceed 200 mg.

### 9.3 Crucible Preparation |

9.3.1 Assemble the filtering apparatus. Insert one filter disk into a crucible with the wrinkled side up and place the crucible in a vacuum apparatus. Wet the filter with a small volume of distilled water to seat it against the support. Turn on the vacuum and wash with three successive 20 mL volumes of DI water.

Continue suction until all traces of water are gone. Turn off the vacuum and place the crucible on the rack. When all crucibles are done, place the rack in a 103-105°C oven for a minimum of one hour.

After removing the rack from the oven, store it in a desiccator until cooled (a minimum of one hour). The moisture content will equilibrate during cooling.

9.3.2        To prepare a crucible for VSS, bring the muffle furnace up to 550 +/- 50°C. Place the prepared gooch(es) into the preheated muffle furnace for 15 minutes. Remove the gooch(es) and allow them to cool to room temperature before putting them into a desiccator. Store the gooch(es) in the desiccator for at least one hour prior to use.

9.3.3        Weigh the crucible and record the weight and crucible number.

9.4        Sample Analysis

9.4.1        Shake the sample vigorously and quantitatively transfer the sample to the filter using a graduated cylinder. Remove all traces of water by continuing to apply vacuum after the sample has passed through.

9.4.2        With suction still on, rinse the graduated cylinder with 10 mL of DI water and add that rinsate to the filter. Apply the vacuum until all the water is drained through. Rinse the filter with 2 additional 10 mL portions of DI water, allowing complete drainage between rinses. Apply suction for about 3 minutes after filtration is complete.

9.4.3        Carefully remove the crucible from the suction apparatus. Dry at least one hour at 103-105°C. Cool in a desiccator and weigh. Repeat the drying cycle until a constant weight is obtained (weight loss is less than 0.5 mg, or less than 4% of the previous weight, whichever is less.)

9.4.4        If VSS is to be determined, transfer the crucibles from 9.4.3 to a muffle furnace that has been pre-heated to 550 +/- 50°C and ignite for 15-20 minutes. Cool somewhat first then cool to the balance temperature in a desiccator. Weigh to the nearest 0.1 mg. Repeat the cycle of igniting, cooling and weighing until the weight loss is less than 4% of the previous weight.



## 9.5 Analytical Sequence

Quality Controls	Frequency	Control Limit
Prep. Blk. (PB)	1 in 20 samples	< Reporting Limit
Lab. Control. Std. (LCS)	1 in 20 samples	+/- 20% Recovery
Lab. Control. Std. Dup. (LCSD)	1 in 20 samples	+/- 20% Recovery
Matrix Spike (MS)	1 in 20 samples	+/- 25% Recovery
Matrix Spike Dup. (MSD)	1 in 20 samples	+/- 20 RPD

►All acronyms are defined in Section 12 of this procedure.

►Drinking water samples must be analyzed in sets of 10 and have a matrix spike and duplicate performed on this matrix. The control limits for LCS's are +/- 10%; Matrix Spikes are +/- 15%; and Matrix Duplicates are ≤ 10 RPD.

## 9.7 Calibration Procedure

Laboratory balances are calibrated and serviced annually by a factory representative. In addition, an analyst checks the balance daily with two masses: one in the gram range and one in the milligram range. A record of calibrations and daily checks will be kept in the balance log.

Class S weights are used by the analysts for daily balance checks.

Oven and refrigerator thermometers will be calibrated annually against a National Bureau of Standards (NBS) certified thermometer in the range of interest. Annual calibrations will be recorded in a calibration notebook. Daily readings will be recorded with the respective oven or refrigerator before and after the drying period.

### 9.7.1 Standards Preparation

#### 9.7.1.1 Preparation Blank (PB)

300 mls DI water

#### 9.7.1.2 Laboratory Control Standard (LCS) and LCS Duplicate; 200 mg/L

300 mLs of Stock Solution I (Rgt. 8.1).



9.7.1.3 Matrix Spike: 25 mg/L

Pour 100 mLs of Stock Solution II (Rgt. 8.2) through the same filter as the sample to be spiked. If possible (see constraints from Section 4.0), spike 100 mL of sample for a spike concentration of 25 mg/L. If other than 100 mL of sample volume was spike, calculate the spike concentration as follows:

$$\text{mg/L spike concentration} = \frac{25 \text{ mg/L} \times 100 \text{ mL}}{\text{mL sample}}$$

NOTE: There is no matrix spike or LCS for VSS. Only a preparation blank and a matrix duplicate can be analyzed.

9.7.1.4 Matrix Duplicate

Duplicate of one sample.

10.0 CALCULATIONS

10.1 Sample Calculation

10.1.1 Total Suspended Solids

$$\text{mg/L} = \frac{(A - B) \times 1,000,000}{C}$$

Where:

A = final weight (g) of crucible plus sample

B = initial weight (g) of crucible

C = sample volume (mL)

10.1.2 Volatile Suspended Solids

$$\text{mg/L} = \frac{(A - D) \times 1,000,000}{C}$$

Where:

A = weight of crucible and residue before igniting (g)

D = weight of crucible and residue after igniting (g)

C = sample volume (mL)

10.2 Accuracy

10.2.1 LCS/LCSD % Recoveries

$$\% R = \frac{\text{observed concentration}}{\text{actual concentration}} \times 100$$

10.2.2 Matrix Spike % Recovery

$$\% R = \frac{(\text{spiked sample}) - (\text{unspiked sample})}{\text{spiked concentration}} \times 100$$

10.3 Precision

10.3.1 Matrix Dup. and LCS Dup. Relative Percent Difference (RPD)

$$\text{RPD} = \frac{|\text{orig. sample value} - \text{dup. sample value}|}{[(\text{orig. sample value} + \text{dup. sample value})/2]} \times 100$$

10.4 Reporting Results

Without rounding, enter the raw data on the appropriate Lotus spreadsheet. Carefully print, review and approve the spreadsheet. Have the data book and spreadsheet approved and signed by the designated reviewer before creating a print file and transferring the data to LIMS.



ANALYTICS DIVISION  
**STANDARD PRACTICES**  
**MANUAL**  
COMPANY CONFIDENTIAL AND PROPRIETARY

**OPERATING PRACTICE**  
**Total and Volatile Suspended**  
**Solids**

Eff. Date: 03/19/93    Initiated By: QC Department    Approved By: D. L. Harper    Authorized By: A. M. Henry    SP No. 21-15G-160.2

11.0        **QUALITY CONTROL**

11.1        One method blank and two Laboratory Control Standards (LCS) will be included in each laboratory lot of 20 samples. Regardless of the matrix being processed, the LCS and method blanks will be in an aqueous media.

11.2        The method blank will be examined to determine if contamination is being introduced in the laboratory.

11.3        The LCS's will be examined to determine both precision and accuracy.

11.4        Accuracy will be measured by the percent recovery (%R) of the LCS. The recovery must be within the laboratory's acceptance limits in order to be considered acceptable. Additionally, %R will be plotted on control charts to monitor method accuracy.

11.5        Precision will be measured by the reproducibility of both LCS's and will be calculated as relative percent difference (%RPD). Results must agree within in-house control limits or statistical control limits in order to be considered acceptable.

11.6        One matrix spike and matrix duplicate is performed per matrix per 20 sample analytical set. Results must agree within the in-house precision/accuracy limits or statistical control limits in order to be considered acceptable.

12.0        **CORRECTIVE ACTIONS**

When an out of control situation occurs, the analysts must use his/her best analytical judgment and available resources to determine the corrective action to be taken. The out of control situation may be caused by more than one variable. The analyst should seek the assistance of his/her immediate supervisor, QA personnel, or other experienced staff if he/she is uncertain of the cause of the out of control situation. The test must not be resumed until the source of the problem and an in-control status is attained. All samples associated with the out of control situation should be reanalyzed. Out of control data must never be released without approval of the supervisor, QA personnel or the lab manager.



12.1 Listed below are steps to be taken when an out of control situation occurs. The analyst must:

- demonstrate that all the problems creating the out of control situation were addressed;
- document the problem and the action which was taken to correct the problem on a corrective action report form;
- document on the corrective action report that an in control has been achieved; and
- receive approval (signature) of the Section Manager, Unit Leader, QA personnel, or the Laboratory Manager prior to the release of any analytical data associated with the problem.

12.2 Suggested Actions to specific out of control situations:

12.2.1 Laboratory Control Standards (LCS)

If either LCS1 or LCS2 exceeds acceptance limits:

- reanalyze LCS to verify that an out of control situation exists;
- determine the source of error within the preparation procedure, correct the problem and repeat the sample set. (Sources of contamination could be either the reagents, the LCS stock solution, or the preparation area.)

Precision: LCS1 and LCS2 must meet the control limits of  $\leq 20\%$  RPD. If this criteria is not met, and both LCS's meet the % Recovery control limits, then see your Section Manager or Unit Leader for proper corrective action.

12.2.2 Preparation Blank (PB)

- reanalyze PB to verify contamination at a level  $>$  Reporting Limit;
- determine the source of contamination and correct the problem;
- all samples whose concentration is  $< 10$  times the PB level must be reprocessed and reanalyzed; any sample which is  $> 10$  times the PB level need not be reanalyzed. However, a corrective action report must be filled out and approval obtained.